Accelerated Testing of Polymeric Composites Using the Dynamic Mechanical Analyzer

B. M. Abdel-Magid, and T. S. Gates

ABSTRACT

Creep properties of IM7/K3B composite material were obtained using three accelerated test methods at elevated temperatures. Results of flexural creep tests using the dynamic mechanical analyzer (DMA) were compared with results of conventional tensile and compression creep tests. The procedures of the three test methods are described and the results are presented. Despite minor differences in the time shift factor of the creep compliance curves, the DMA results compared favorably with the results from the tensile and compressive creep tests. Some insight is given into establishing correlations between creep compliance in flexure and creep compliance in tension and compression. It is shown that with careful consideration of the limitations of flexure creep, a viable and reliable accelerated test procedure can be developed using the DMA to obtain the viscoelastic properties of composites in extreme environments.

INTRODUCTION

The long-term exposure of polymeric composite materials to extreme-use environments, such as pressure, temperature, moisture, and load cycles, results in changes in the original properties of the material. These changes in material properties translate to structural changes that can have a potentially catastrophic effect on load-bearing composite structures. Therefore the study and understanding of the long-term effect of exposure on the time-dependent viscoelastic properties of polymeric composite structures is crucial to their proper design, construction, and safe operation. Two experimental options are used to characterize the viscoelastic properties of polymeric composites: the conventional and time-consuming method of directly measuring the response over long periods of time, and the second method of using accelerated aging processes to obtain the properties during short-term testing.

Numerous investigators have used the DMA to study the viscoelastic behavior of polymer matrix composites [1,2,3,4]. The advantages of using the DMA to characterize the viscoelastic creep properties of these materials include:

Beckry M. Abdel-Magid, Winona State University, P.O. Box 5838, Winona, MN 55987
Thomas S. Gates, NASA Langley Research Center, MS 188E, Hampton, VA 23681

automated testing, better control of the test environment, simple preparation of test specimens, and performing of creep test at higher, above T_g, temperatures. However, the flexure-loading mode used in the DMA specimens introduces difficulties of isolating tensile properties from compressive properties of the material.

In this study an accelerated method is developed using the dynamic mechanical analyzer (DMA) to measure the viscoelastic creep properties of polymeric composite materials. The objectives of the study are to: investigate the use of the DMA in finding creep properties of polymeric composite materials, compare results from DMA creep tests with data from conventional creep tests, assess the accuracy of results of the DMA sub-coupon level tests, and investigate the potential of using the DMA results to predict the long-term creep properties of composite materials.

In this study, three experimental procedures were used to compare the results of the DMA testing with pure tension and pure compression behavior. They were DMA flexure creep, tensile creep and compressive creep. Creep compliance curves and time temperature superposition (TTSP) master curves were obtained from each test. The master curves and the resulting shift factors are compared in this study. Theoretical background, test procedures and the analysis of results are presented in the following sections.

THEORETICAL BACKGROUND

The time-dependent linear creep compliance was modeled as:

$$S(t) = S^0 e^{(t/\tau)^{\beta}} \tag{1}$$

where S^0, τ, β are the initial compliance, retardation time, and shape parameters, respectively [5,6].

The use of time-temperature superposition (TTSP) [5] requires that creep compliance be a function of temperature (T) and time (t), such that:

$$S = S(T, t) \tag{2}$$

and that:

$$S(T, t) = S(T_0, \varsigma) \tag{3}$$

$$\varsigma = t / a_T(T) \tag{4}$$

where ς is the reduced time that is related to the real time t by the temperature shift factor $a_T(T)$, and T_0 is the reference temperature. Further details of the model can be found in [5].

EXPERIMENTAL PROCEDURES

To utilize the previous expressions, creep compliance curves were obtained at a series of temperatures over a specific time period and a single curve at a specified temperature was selected as the reference curve. The specified temperature was based on the use environment. Using TTSP all the curves were shifted horizontally along the log-time scale until they superimposed on the reference curve creating the master curve. Characterizing the master curve with equation (1) allowed the determination of creep compliance as a function of time. In general, complete analysis of high temperature creep should include the physical aging effects due to elevated temperatures in the construction of master curves [6,7]. However in this study, the effect of physical aging is accounted for by using equivalent thermal history in all three test programs. The master curves and the TTSP shift factors were determined experimentally for the three test conditions.

Materials and Test Specimens

The material system chosen for this study was a unidirectional carbon/thermoplastic polyimide composite designated as IM7/K3B. The glass transition temperature, $T_{g,}$ of the material was 240°C. Test specimens were cut from laminated panels ranging in thickness from 1.7 mm to 1.775 mm. The flexure specimens were prepared according to ASTM Specification D790 measuring 60 mm by 9.4 mm in size. Tension and compression specimens were similar in size to those described in ASTM D3039, measuring 240 mm by 19 mm for tension tests and 200 mm by 19 mm for compression tests. The specimens were loaded perpendicular to the fibers to determine the viscoelastic creep properties in the matrix-dominated transverse direction. To reduce experimental errors, at least three replicates were tested at each creep sequence in each of the three test programs. Prior to testing, specimens were dried for 24 hours at 110°C.

DMA Flexure Creep

LINEARITY

To ensure that the tests were performed within the linear viscoelastic range, a preliminary study was conducted to check that proportionality conditions and Boltzmann's superposition would be satisfied [5]. A DMA specimen was subjected to sets of creep and creep recovery sequences at flexural stresses ranging from 3.4 MPa to 4.8 MPa with increments of 0.2 MPa at the reference temperature of 225°C. After each test, the specimen was rejuvenated, quenched and loaded at the next stress level. The rejuvenation process is described by Struik and others [6,7]. These tests provided data for checking superposition. Proportionality checks were made by plotting the creep compliance versus time at each stress level. No significant vertical separation between the compliance curves was observed indicating that there was no transition from linear to nonlinear behavior in the applied stress range.

CREEP SEQUENCE

Specimens were tested below the glass transition temperature of the material, and were subjected to a sequence of thirty-minute load and recovery steps. A constant stress of 4.2 MPa was applied for thirty minutes during which deformation was measured. The load was then removed and the specimen was allowed to recover for thirty minutes. After recovery the temperature was increased by 5°C and the specimen was allowed to equilibrate for 10 minutes at each temperature step. Temperatures between 210°C and 230°C were used. A schematic of the sequenced creep-recovery procedure is shown in Figure 1 along with the desired properties.

In the flexure test the maximum deformation, δ, at the center of the beam was measured with time. The strain is determined as:

$$\varepsilon_{\max}(t) = \frac{6d\delta(t)}{L^2\left[1+\frac{6}{10}(1+\nu)(\frac{d}{L})^2\right]} \quad (5)$$

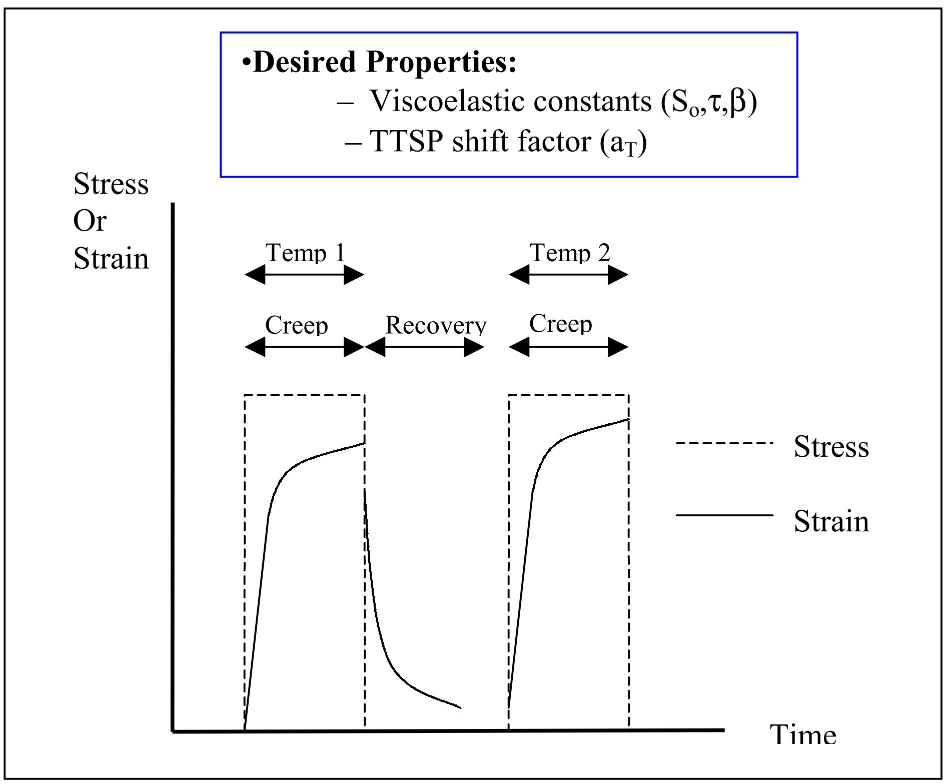


Figure 1. Creep Sequenced Test Procedure

where t is time, ν is the material's Poisson's ratio d is the specimen thickness and δ, and L are shown in Figure 2. A description of the DMA 3-point bend creep test is shown in Figure 2 with the corresponding test parameters and equations for strain, stress, and creep compliance.

Tension and Compression Creep Tests

The creep and recovery sequence described above was used in the tension and compression creep tests. These tests were conducted on creep-test equipment and fixtures developed in previous work by Gates and Veazie [6,8]. Each specimen was instrumented with two back-to-back strain gages and the axial load was applied through a dead-weight cantilever arm system. The tensile specimens were subjected to a constant stress of 2.11 MPa and the compression specimens were subjected to stress of 2.57 MPa. Linearity checks for these loading conditions were conducted in a previous study by Gates et al. [6]. After each creep sequence the specimens were rejuvenated and the tests were repeated at least three times. A schematic of the tension and compression creep tests along with the test parameters are given in Figure 3.

The effect of physical aging on creep behavior of IM7/K3B composite material was investigated in a previous study by Gates and Feldman [9]. By keeping the same creep sequence and thermal history in all tests, the specimens were subjected to the same physical aging effects. Since this is a comparative study, the aging effects on the specimens were not considered in the analysis of results.

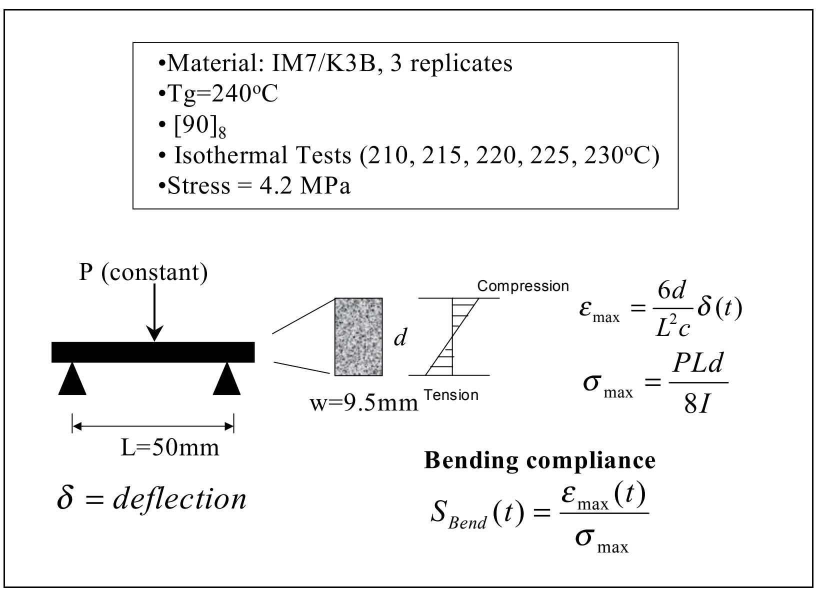


Figure 2. DMA 3-Point Bend Creep

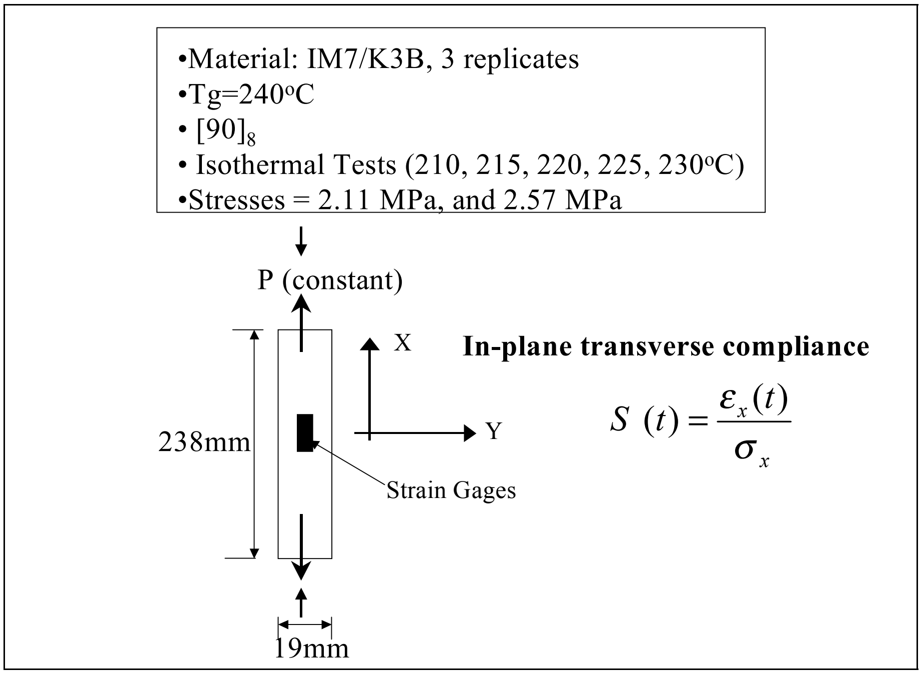


Figure 3. Uniaxial Tension and Compression Creep

RESULTS AND DISCUSSION

Creep Compliance Curves

Creep compliance, S, versus time, t, curves were obtained for the five test temperatures of 210°C, 215°C, 220°C, 225°C, and 230°C, with stress duration of thirty minutes each. These curves were obtained using the three test procedures of flexure, tension, and compression creep. The creep compliance curves of the DMA flexure tests are given in a log-log scale in Figure 4. This figure shows that creep compliance of the IM7/K3B composite was a function of test temperature, with an increase in temperature resulting in an increase in both compliance and creep rate. The highest compliance was obtained at the temperature of 230°C at the end of the half-hour creep period. Similar curves were obtained from the tension and compression creep tests.

TTSP Master Curves

A reference temperature of 225°C was selected for this study, and the principle of time-temperature superposition was used to shift the compliance curves and obtain the master curve at the reference temperature. Master curves of the DMA flexure tests, tension tests, and compression tests are given in Figures 5, 6, and 7, respectively. The master curves shown in these figures cover an expanded

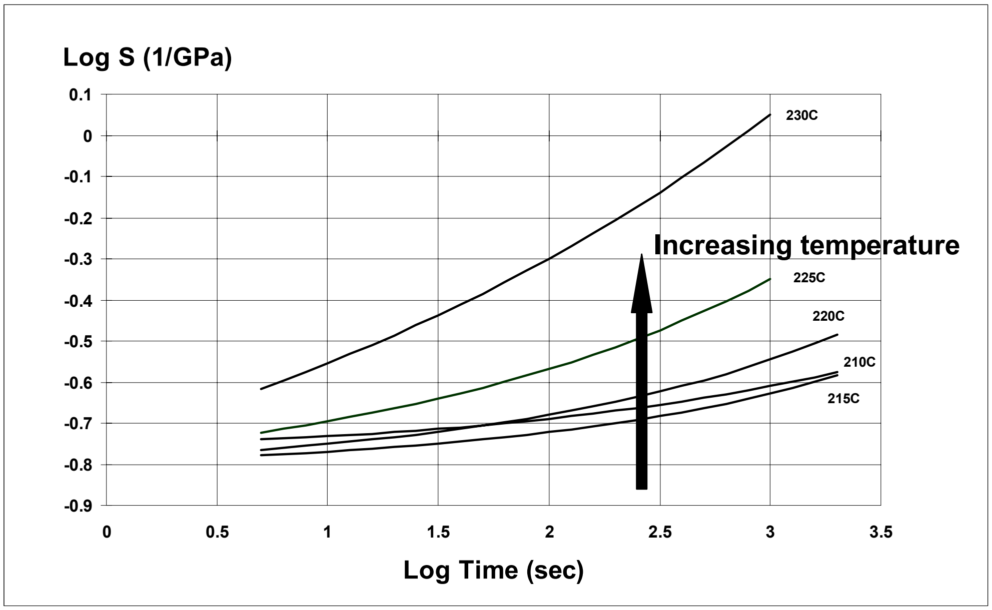


Figure 4. DMA Flexure Creep Compliance Curves

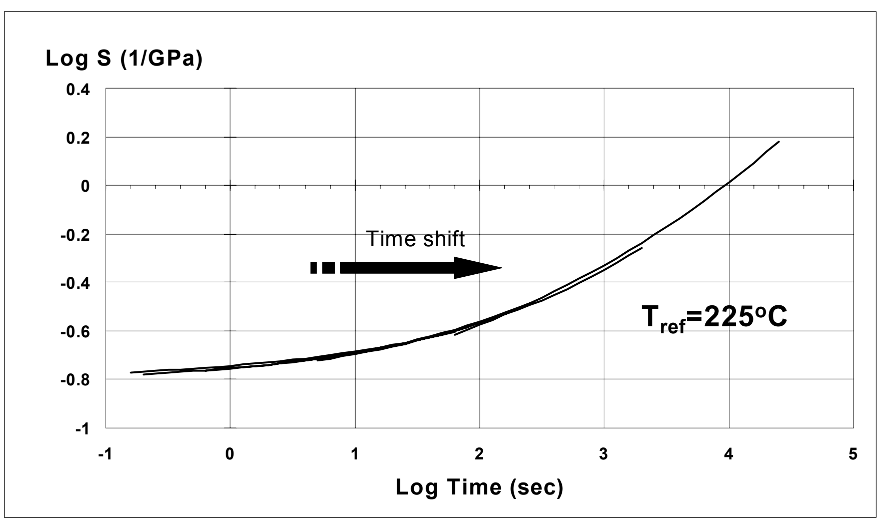


Figure 5. DMA Creep Compliance TTSP Master Curve

Figure 6. Tensile Creep Compliance TTSP Master Curve

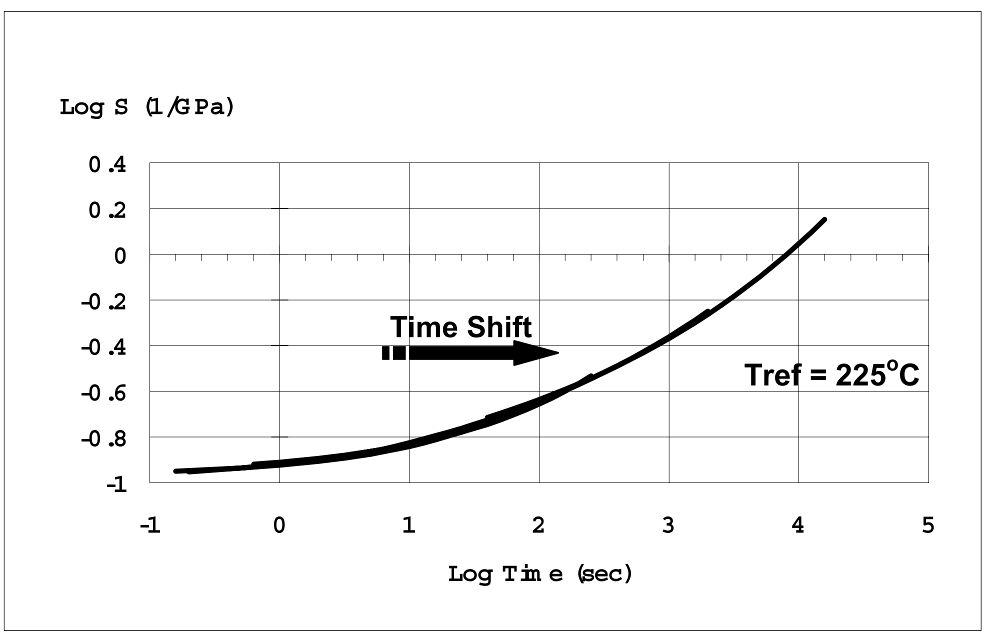


Figure 7. Compression Creep Compliance TTSP Master Curve

period of time reaching 4.4 hours as a result of collapsing the thirty-minute creep compliance curves to the reference temperature of 225°C.

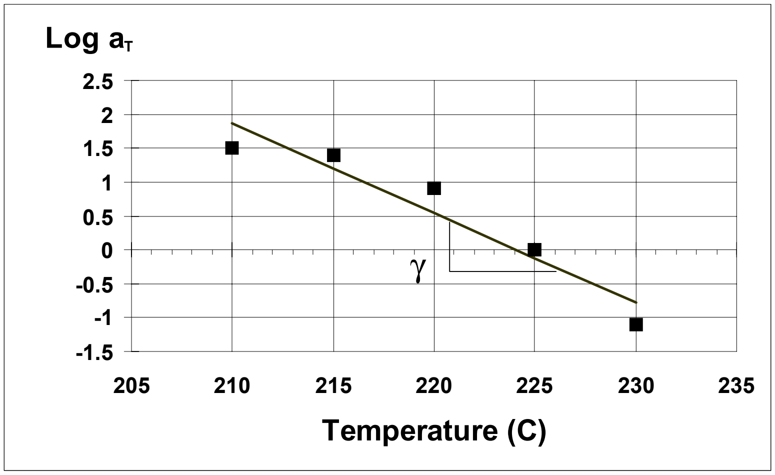
The master curves shown in these figures exhibit slight differences between them. Examination of Figure 7 reveals that initially the creep curves for the compression-loaded specimens shifted lower on the compliance axis than the corresponding tension-loaded specimens given in Figure 6. On the other end of the spectrum, for extended duration of creep, the compression loaded specimens exhibited higher compliance than the tension-loaded specimens. This is indicated by the higher curvature at the top of the master curve in Figure 7 compared with the master curve in Figure 6. The difference in these master curves indicates that the loading mode, tension or compression, influences the creep behavior of the IM7/K3B composite. Similar observations were reported by Gates et al. [6].

Examination of Figure 5 reveals that the flexure master curve is also slightly different from both the tension and compression master curves. The flexure compliance curves shift lower than the tension curves at the initial part of the master curve, and they shift higher than the tension curves at the upper part of the master curve. The flexure master curve essentially lays in the middle between the tension master curve and the compression master curve. This is a further indication that the mode of loading influences the creep behavior of the composite material. This influence was shown in previous studies to be more prominent when predictive models are used to determine creep compliance at extended periods of time [6,9].

TTSP Shift Factors

Shift factors, a_T, were determined for each master curve. The data are plotted as $\log a_T$ versus temperature in Figures 8, 9, and 10 for flexure, tension and compression creep, respectively. Each curve indicates the magnitude of the total shift in the corresponding master curve. In these figures the $\log a_T$ versus temperature curves are approximated by straight lines. At the reference temperature, $\log a_T$ is zero, that is the shift factor $a_T = 1$. The slope of the straight line is the shift rate, γ. The shift rate can be used in predictive models [5] to determine the creep compliance as a function of time.

The values of the shift rate for each master curve are given in Figures 8, 9, and 10 for flexure, tension and compression master curves, respectively. These values are the calculated mean of three tests in each test type. The shift rate for the DMA flexure creep master curve was determined to be -0.132. For the tension and compression master curves, γ was found to be -0.091 and -0.122, respectively. The differences in the values of the shift rate may also reflect the influence of the loading mode on the creep behavior. The shift rate obtained from the flexure test was closer to the shift rate obtained from the compression test, and was not radically different from the shift rate obtained from the tension test. These results indicate that the DMA flexure creep test closely resembles the tension and compression creep behavior of polymeric matrix composites.

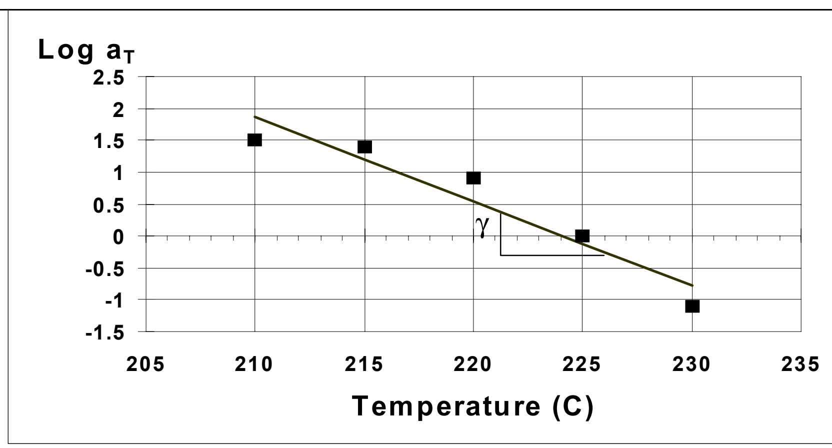


a_T Shift Factor

$$\gamma = \frac{d \log a_T}{dT}$$ Shift Rate: Used in predictive model

$\gamma = -0.132$ for 3 pt. Bend creep

Figure 8. DMA Creep Compliance TTSP Shift Factors

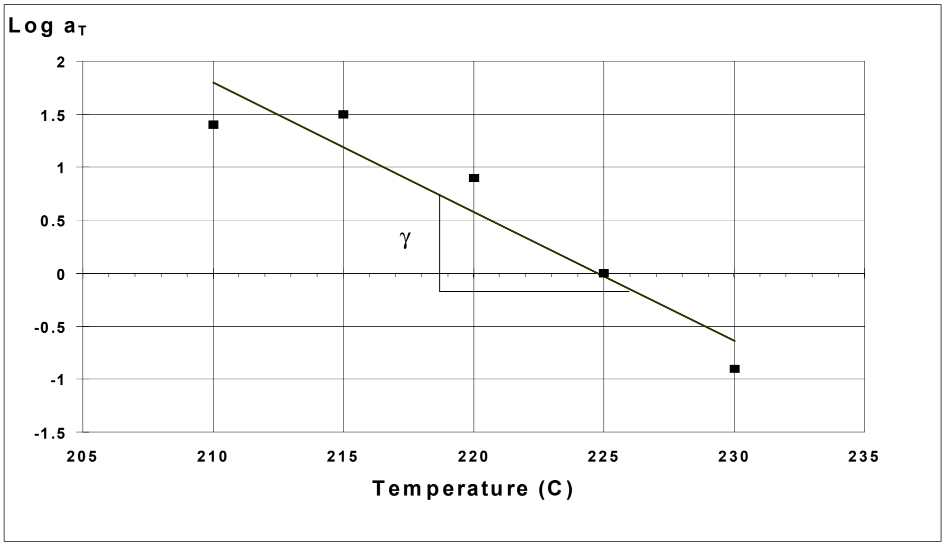


a_T Shift Factor

$$\gamma = \frac{d \log a_T}{dT}$$ Shift Rate: Used in predictive model

$\gamma = -0.091$ for Tensile creep

Figure 9. Tensile Creep Compliance TTSP Shift Factors



a_T Shift Factor

$\gamma = \dfrac{d \log a_T}{dT}$ Shift Rate: Used in predictive model

$\gamma = -0.122$ for Compression creep

Figure 10. Compression Creep Compliance TTSP Shift Factors

SUMMARY AND CONCLUSIONS

An experimental study was performed to investigate the use of DMA flexure testing in predicting the long-term creep behavior of polymeric matrix composites. Creep compliance curves and TTSP master curves were obtained from DMA flexure creep tests, tensile creep tests and compression creep tests. The tests were performed on IM7/K3B composite under similar stresses and identical thermal history conditions to eliminate differences in the physical aging effects on the material. The composite was tested at sub-T_g temperatures. Three replicates were used in each test and the data were consistent and repeatable over the entire range of test temperatures.

Comparison of results from the three test programs indicated that the loading mode caused a slight difference in the creep behavior of polymer matrix composites. It was shown that while the DMA flexure creep results were not identical to the tension and compression creep results, the DMA test closely resembled the tension and compression creep behavior of polymeric matrix composites. Furthermore, the results confirmed a previous finding that the slight difference between flexure, tension and compression creep may reflect a real change in material behavior under these modes of loading.

In spite of the combined loading modes (tension, compression and shear) in the DMA flexure test, the consistency in the data indicates that a high level of accuracy was exhibited in the test results. To establish further confidence in the accelerated DMA creep test, it is recommended that the effects of physical aging of the composite during the DMA test be investigated. It is also recommended that the shear effect in the 3-point bend DMA test, especially at elevated temperatures, be investigated.

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REFERENCES

1. McMaster, A. D. and D. Chundury. 1994. "Creep Data for Gapex Polypropylene Products via DMA Method," presented at the SPE 52nd Annual Technical Conference, May 1-5, 1994.
2. Sepe, M. P. 1998. *Dynamic Mechanical Analysis for Plastic Engineers*, PDL Handbook Series, Plastic Design Library.
3. Wen, V. F., R. F. Gibson, and J. L. Sullivan. 1995. "Characterization of Creep Behavior of Polymer Composites by the Use of Dynamic Test Methods," presented at the 1995 ASME International Mechanical Engineering Congress and Exposition, Nov. 12-17, 1995, Vol. 20, pp. 383-396.
4. Wong, C. P., Y. Rao, and J. Qu. 1998. "Creep Behavior Characterization of Some New Materials for High Density Interconnect Substrates Using Dynamic Mechanical Analyzer (DMA)," presented at the 4th International Symposium on Advanced Packaging Materials, March 15-18, 1998, pp. 73-76.
5. Findley, W. N. J. S. Lai, and K. Onaran. 1976. *Creep and Relaxation of Nonlinear Viscoelastic Materials*. Toronto; North Holland Publishing Company, pp. 103-107.
6. Gates, T. S., D. R. Veazie, and L. C. Brinson. 1997. "Creep and Physical Aging in a Polymeric Composite: Comparison of Tension and Compression," *J. Composites Materials*, 31 (24): 2478-2505.
7. Struik, L. C. E. 1978. *Physical Aging in Amorphous Polymers and Other Materials*. New York; Elsevier Scientific Publishing Company.
8. Veazie D. R. and T. S. Gates. 1995. "Physical Aging Effects on the Compressive Linear Viscoelastic Creep of IM7/K3B Composite," NASA, TM-110224, Hampton, VA.
9. Gates, T. S. and M. Feldman. 1996. "Effects of Physical Aging at Elevated Temperatures on the Viscoelastic Creep of IM7/K3B," in *Composite Materials: Testing and Design (Twelfth Volume), ASTM STP 1274*, R. B. Deo and C. R. Saff, eds., American Society for Testing and Materials, 1996, pp. 7-36.